Determination by infrared spectroscopy of triacetin content in diesel: A tool for countering designer fuel frauds

Rita Pastres [a], Anna Luisa Panzeri [b,*], Doriana Visentin [b], Valerio Causin [a,*]

[a] Dipartimento di Scienze Chimiche, Università di Padova, via Marzolo 1, Padova 35131, Italy
[b] ADM Agenzia delle Dogane e dei Monopoli, Ufficio Antifrode e Controlli – Sezione Laboratori, Laboratorio di Venezia, DTIII -Veneto e Friuli Venezia Giulia, via dell'Elettricità, 19, Venezia 30175, Italy

ABSTRACT

Taxation on fuels is an important share of fiscal revenues of most countries. Among the most abundant petrochemicals in the market, gas oil, or diesel, is particularly important, and it is commonly used as fuel for heating systems and for vehicles such as cars and trucks, but also for farm machinery and boats. Due to its wide range of applications and to the fact that most of the final price of diesel is made up of taxes, the phenomenon of designer fuels is increasingly common. Designer fuels are fuels in which some chemical and physical characteristics are fraudulently modified in order to classify them as other goods, e.g. lubricating oils or solvents, which are subject to lower or no taxes.

Triacetin is a frequently employed additive for changing the classification of heavy distillates into lubricant oils, and it is therefore important, for a customs office, to be able to assess its presence as quickly and easily as possible, both inside or outside the laboratory. This paper proposes a method based on IR spectroscopy for the detection and quantification of triacetin in diesel, in an inexpensive but effective way. The analytical protocol presented allows a limit of quantification as low as 0.3% v/v, it is robust, precise and accurate enough to yield data useful for the customs agent to take decisions without further lengthy procedures.

1. Introduction

Petroleum products are divided into heavy, medium or light according to the number of carbon atoms in the hydrocarbon molecules and their distillation range. Gas oil is a medium-heavy fraction of petroleum, obtained by fractional distillation, and chemically it is a mixture of aliphatic, cyclic and aromatic hydrocarbons composed of 12 to 25 carbon atoms. Gas oil has a distillation range from 170 to 360 °C, which partially overlaps that of adjacent fractions: kerosene on the light side of the range and heavier lubricants or fuel oils on the heavy side. Due to the ample variability in applications and performance requirements, petroleum products cannot be classified solely according their distillation range, but it is necessary to consider other parameters [1].

Due to its prominent position, along with gasoline, among automotive fuels, diesel is subject to taxes and regulations which determine its compliance with environmental laws and its suitability for use in vehicles. This complex legislative framework shaped the typical analytical procedure used to characterize diesel [1,2 and references therein].

Features such as cetane number, density and viscosity, content of polycyclic aromatic hydrocarbons, sulphur, manganese, ash and water, flash point and distillation range are usually determined in order to classify a petrochemical as diesel. Fatty Acid Methyl Alkyl Esters (FAMAE) content is another key parameter, because up to 7.0% (v/v) of FAMAE is allowed in commercial diesel, although different countries can apply different limits. Moreover, dyes and markers allow to further categorize diesel according to its legitimate use. Many countries, in fact, apply a favorable taxation to gas oil used in farm machinery, fishing boats or for heating. Obviously, using rebated fuels for different purposes than those prescribed is illegal, so it is forensically important to be able to identify what category a certain gas oil pertains to.

Two different approaches for the taxation of petroleum products are generally adopted by national fiscal authorities. One is excise taxes, which are imposed on a good when it is produced or bought by a wholesaler. The other is consumption tax, which is paid when a particular good is used. Taxation on goods is applied according to their Harmonised System / Combined Nomenclature code. The International Convention on the Harmonized Commodity Description and Coding

System (HS Convention) entered into force on 1 January 1988, with the aim of facilitating international trade by harmonizing the description, classification and coding of goods in international trade. The Combined Nomenclature (NC) [3] is a further development, with special European Union (EU)-specific subdivisions, of the World Customs Organization's Harmonized System nomenclature, which is used to classify most goods when they are declared to customs in the EU.

If the analysis of a product reveals anomalies with respect to the chemical-physical characteristics associated to its HS/NC code, the product is declared "non-compliant". In the case of diesel fuel, a non-compliant product could, for example, be a blend with concentrations of over 7% v/v FAMAE or with an excessive sulphur content. A discrepancy with the declared product does not always constitute a fraud, because this term usually refers to a deliberate attempt to evade or circumvent a binding regulatory provision, thereby making a financial profit and avoiding, for instance, payment of excise duty, consumption tax or value-added tax.

Aside from the addiction of subsidized products with characteristics compatible with those of automotive diesel, adulteration with solvents or other industrial products is a widely used fraudulent strategy for gas oil.

Fraud, and smuggling in the energy products sector in general, is a growing phenomenon and customs laboratories are faced with the need to quickly and early identify cases of illegality in this area. Currently, in Italy fraud due to the so-called "designer fuels" is frequent. These are diesel fuels, declared as lubricating oils with documents which indicate that they are only in transit through Italy. Their declared origin and destination countries are frequently non-EU countries. However, these goods do not travel as stated, but they are distributed for consumption in Italy with a subsequent non-payment of taxes. The mechanism of this scheme is based on the fact that diesel and also fuel oils are subject to excises, lubricants to consumption taxes. The overall effect of the fraudulent mechanism is the transit between countries of a product classifiable as lubricating oil (not subject to excise duty harmonized at the EU level) which, at the time chosen, is used as regular diesel, with complete avoidance of tax payment.

Although the lately adopted administrative tracing methods are effective tools against smuggling and contraband of fuels, chemical analysis remains the primary and basic strategy against the "designer fuels" phenomenon.

These designer fuels have distillation characteristics very close, even though not perfectly superimposable, to those of diesel: they have the same sulphur content, the same viscosity and a compatible IR spectrum, except for small differences due to the presence of polar compounds that modify the saponification number parameter. This is the quantity of strong base, expressed in mg, required to saponify one gram of fat and to neutralize free fatty acids [4]. In the viscosity range of the petroleum products of interest in this work, the threshold between fuel oils and lubricants is 4 mg KOH/g [5]. Anomalies in the distillation, i.e. the failure to recover 85% v/v including losses at 350 °C, do not allow the classification of these products as diesel, but makes them identifiable as fuel oils or lubricants, on the basis of the relative combination of viscosity, color and saponification number. This latter parameter is indeed the most discriminating in the classification of these case. Addition of triacetin is a common way to increase the saponification number of diesel, shifting its classification towards lubricating oil, and therefore allowing to elude taxation.

Triacetin is the common name of glyceryl triacetate, an ester of acetic acid and glycerol. It is generally used as a humectant in the food industry, in the pharmaceutical industry, and as a plasticizer in the production of cigarette filters. Triacetin is a by-product of the interesterification reaction between triglycerides and methyl acetate and it has a higher added value than glycerol [6,7]. For this reason, recent research in the field of FAMAE is focusing on improving interesterification processes and on testing triacetine as an additive for FAMAE [6–16].

Even though mixed results are reported in the literature about its suitability as an additive for diesel, it is clear that triacetin-containing heavy distillates maintain many of the chemical-physical characteristics suitable for use as diesel, including cetane number.

On the other hand, small quantities of this compound to diesel-like fuels can efficiently increase the saponification number to values exceeding 4 mg KOH/g, allowing the classification of such materials as lubricating oils avoiding the classification as fuel oil, subject to harmonised excise duty within the EU. Two other things are worth noting. Firstly, the 4 mg KOH/g limit forthe saponification number does not apply to products containing one or more bio-components, i.e. animal or vegetable fats, animal or vegetable oils, or FAMAE. Under this definition, triacetin does not qualify as a biocomponent, so it is indeed key for changing the classification of a diesel. Secondly, it is easy to convert a triacetin-containing designer fuel into regular diesel by mixing with legitimate fractions just before putting it into the market. This can avoid undesirable effects and damage to engines that can be easily detected during use, and that could bring about a negative quality judgment by the costumer.

The aim of this work was to set up a quick and straightforward method for the quantification of triacetin present in petroleum products declared as lubricants, but suspected to be disguised diesel. Fourier Transform-Infrared (FT-IR) spectroscopy was chosen for this objective, because of its wide availability, low cost, short analysis times and ease of use. Correlating triacetin content with saponification number could allow to avoid lengthy retro-titration procedures and also to put the premises for a roadside analysis protocol, where trained police officers can test a suspect product and take decisions on its legitimacy.

2. Materials and methods

Diesel fuel is a complex matrix that is difficult to reproduce with a solvent mixture. Two sets of standards were prepared: one diesel without FAMAE and one with less than 7% v/v of FAMAE. Triacetin (99%) was purchased from Sigma-Aldrich (CAS 102-76-1). From a first series of tests, it was noted that a 3% v/v solution of triacetin in diesel shows phase separation; this is due to the fact that triacetin, because of its polar groups, has limited miscibility with diesel hydrocarbons. For calibration, solutions of known concentration of triacetin were prepared for each matrix: 1.5, 1, 0.75, 0.5, 0.3, 0.12% v/v.

For comparison and validation purposes, 7 samples seized in previous anti-fraud operations were used. For each of these samples a complete characterization according to the technical norm EN 590:2013+A1:2017 [1] was made, also quantifying saponification number and polar extract fraction.

Infrared (IR) absorption spectra were acquired on a PerkinElmer Frontier FT-IR Spectrophotometer equipped with an attenuated total reflection (ATR) accessory with a single reflection diamond. The spectral region spanned from 4000 to 650 cm^{-1}. Three 16-scan spectra were collected for each solution, and the spectra were averaged. The measurement uncertainty was estimated as maximum semi-dispersion. The error on the concentration of the standard solutions was assumed to be negligible. Neither water/CO_2 correction nor ATR correction were applied on the IR spectra. A background scan was acquired before every sample, in order to cancel the effect of the atmosphere on spectral quality. It is known that ATR introduces a distortion in peak intensities, because penetration depth of the evanescent wave, and thus the intensity of absorption bands, increases linearly with wavelength. Another known artefact introduced by ATR is a shift of absorption bands to lower frequencies. However, these may be relevant issues just when searches in databases are involved. In this case, all spectra were acquired in the same conditions, and so, even if the instrument software allowed to correct the spectra for ATR distortions, it was preferred to use raw data for the analysis, avoiding the potential introduction of artefacts due to correction algorithms. Spectra are displayed in log(1/R) units, where R is reflectance, which are dimensionally equivalent to absorbance [17]

Robustness was assessed modifying experimental conditions and comparing the obtained results. The only significant method parameter which could be changed was the number of scans, so the most and the less concentrated standard samples were acquired with 8, 16, 32, 64 and 128 scans. The dependence of results from the used instruments was estimated by constructing the calibration curves with a different instrument: FTIR Nicolet 5700 spectrometer (Thermo Nicolet) in ATR mode with a Thermo ATR ITX (Thermo) equipped with a diamond ATR crystal.

Nicolet Omnic software (rel. 6.0a) was used for the treatment of spectra. Baseline correction was not applied in any measurement procedure. Integration of the peaks was performed, on the spectra in log(1/ R) units, using the same integration limits for all the samples. These limits were chosen to encompass about 95% of the total area, excluding the band edges [18]. Peak heights were measured from the maximum of the peak to the baseline traced through the minima on each side of the absorption band. If outliers were encountered in the data, they were dropped and the measurement was repeated.

Cetane number was estimated according to ASTM standard D7170-16 [19].

Quantification of triacetin in the reference samples for comparison purposes was made with an in-house method by SPE extraction and gravimetric determination followed by gas chromatography-mass spectrometry identification [20]. In particular, polar extracts were obtained gravimetrically by extraction through a silica gel (Pore size 60 Å, particle size ~60–200 μm, mesh size 70–230).

Saponification number was determined according to ASTM D94-07 (2017) [4].

3. Results and discussion

Diesel fuel is a mostly saturated hydrocarbon mixture that contains a maximum of 7% v/v of FAMAE Fig. 1. shows the spectra of two samples of diesel fuel: the first without FAMAE and the second with a certain amount of it, less than 7% v/v.

In both spectra, the characteristic vibrations of normal-alkanes are visible: the three peaks in the $2900 \ cm^{-1}$ region are related to the stretching of CH bonds in CH_2 and CH_3 groups, while the two peaks at $1400 \ cm^{-1}$ are attributed to the bending of the same groups. The presence of the aromatics is also visible, in particular C-C stretching in the $1600–1585 \ cm^{-1}$ and $1500–1400 \ cm^{-1}$ region and aromatic CH stretching around $3000 \ cm^{-1}$ are noted. The bending in and out of the plane of aromatic CH groups between 900 and $675 \ cm^{-1}$ and

$1300–1000 \ cm^{-1}$ complete the description.

The only difference between the spectra is the medium-intensity peak at wavenumber of $1747 \ cm^{-1}$ in Fig. 1b; the band is due to stretching of the carboxyl group –C=O, which is due to the methylesters of FAMAE and does not appear in a hydrocarbon-only mixture such as diesel.

Triacetin can be easily detected by FT-IR spectroscopy Fig. 2.a shows the IR spectrum of pure triacetin. As may be seen, the two most prominent absorptions are located at $1755 \ cm^{-1}$ and at $1218 \ cm^{-1}$.

As may be seen, triacetin adds to both types of considered diesel fuel a peak at about $1750 \ cm^{-1}$, due to its carboxylic groups. However, this cannot be considered as a diagnostic peak, because it overlaps the signal in the same position due to the C=O groups of FAMAE. However, the peak at $1218 \ cm^{-1}$ is characteristic and unique for triacetin; from a comparison of the IR spectra in Figs. 1 and 2, it can be seen that this peak is not only indicative of the presence of triacetin, but it is absent in compliant diesel with or without FAMAE and distinguishable even from diesel sophisticated with oxo oils, thus making the method selective precisely for the sophistication approach of interest in this work.

Therefore, the peaks at $1218 \ cm^{-1}$ were quantified in the two available sets of standard solutions. Three spectral quantification approaches were explored in the data analysis phase: peak height of the $1218 \ cm^{-1}$ signal, area of the same peak and ratio of the area of the peak at $1218 \ cm^{-1}$ and of the area of the diesel-specific peak centered at approximately $1450 \ cm^{-1}$. The best linearity in calibration curves was obtained by plotting the peak height data as a function of triacetin content. For this reason, this will be the only approach reported in the rest of this work; the areas were more dependent on a somewhat subjective choice of integration range and also were more affected by the signal-to-noise ratio provided by the instrument.

The spectra of the standard solutions without FAMAE are shown in Fig. 3 to highlight the variation of the peak under analysis at $1218 \ cm^{-1}$. As may be seen, the peak is barely detectable in the sample with 0.12% triacetin, whereas at higher concentrations, the absorption can be seen and integrated.

Fig. 4 shows the calibration curves obtained on both matrices. The curve that best interpolates the data in both cases is a straight line, whose parameters are reported in Table 1. R^2 values are >0.999 in both cases. In the case of the matrix not containing FAMAE, the sample with the highest triacetin content, i.e.1.5%, showed a significant deviation from linearity (Figure S1 in the supporting information) and was therefore not considered for constructing the calibration curve. On the other hand, the standards containing FAMAE retained their linearity
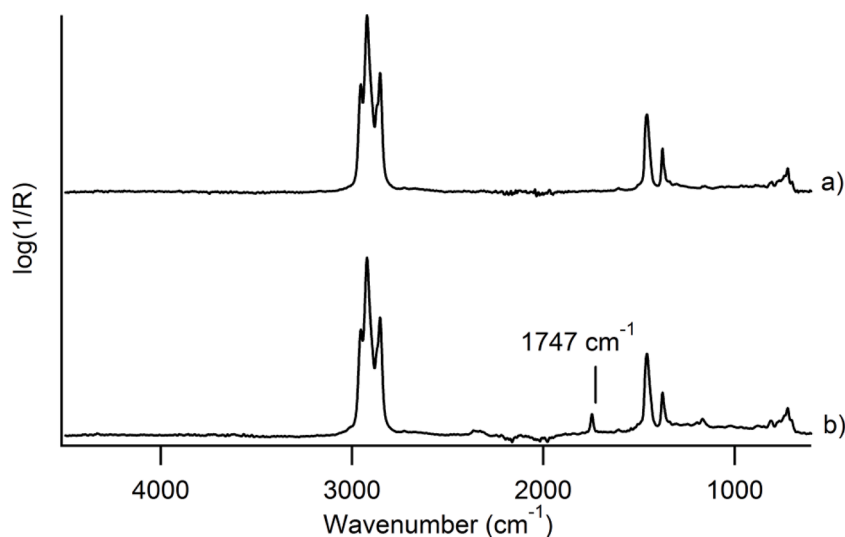

Fig. 1. IR spectra of (a) diesel without FAMAE and (b) diesel with FAMAE. The position of the FAMAE C=O stretching peak is indicated. Spectra are offset on the y axis for clarity.
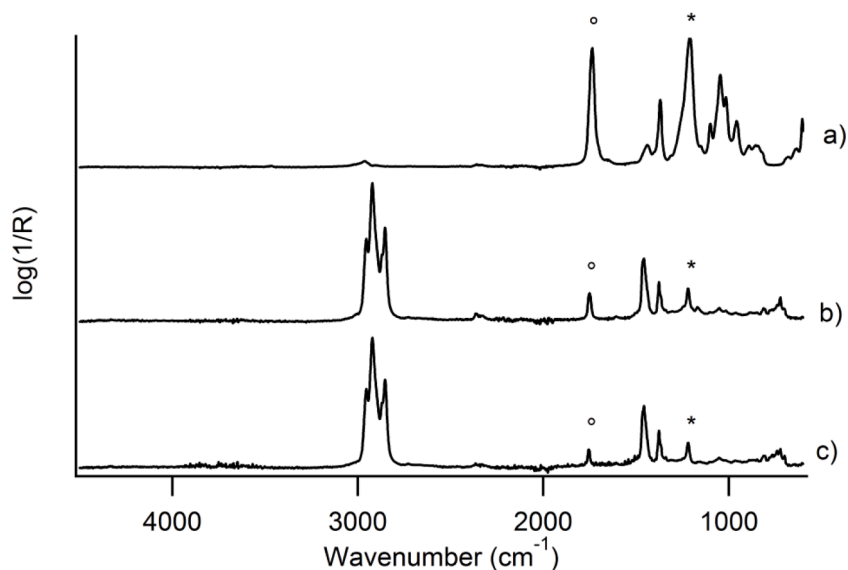
Fig. 2. IR spectra of (a) triacetin, (b) diesel without FAMAE containing 1.5% v/v triacetin and (c) diesel with FAMAE containing 1.5% v/v triacetin. The ° and * labels indicate the two most prominent absorptions of triacetin, located at 1755 cm^{-1} and at 1218 cm^{-1}, respectively. Spectra are offset on the y axis for clarity.
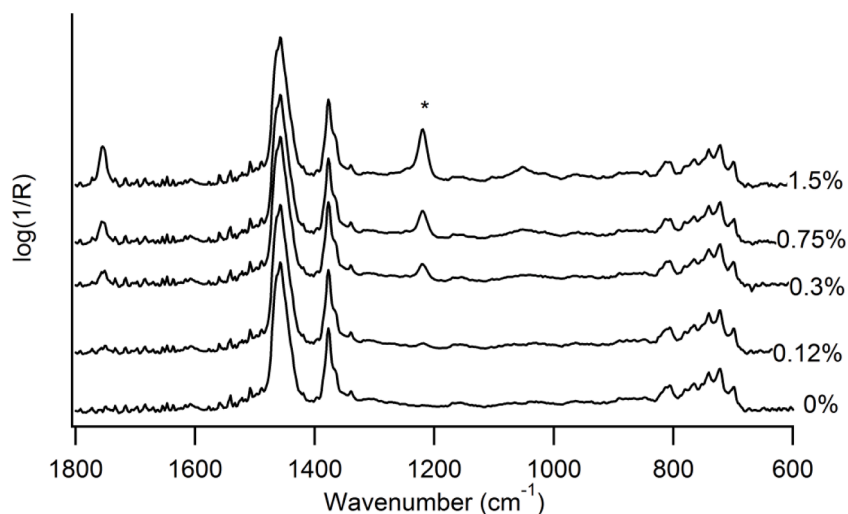

Fig. 3. IR spectra of a matrix of diesel without FAMAE with different concentrations of triacetin. The * indicates the position of the diagnostic peak of triacetin, located at 1218 cm^{-1}. Spectra are offset on the y axis for clarity.

also in the most concentrated sample. This is an indication of a significant matrix effect, which has been also quantitatively detected (*vide infra*).

It is worth noting that both curves have an intercept significantly different from zero. This is consistent with a very low limit of detection, but with a limit of quantification not lower than 0.3% v/v triacetin. In other words, at very low triacetin concentrations, quantification is not reliable and just the presence/absence of this additive can be concluded.

A Student's *t*-test at 95% confidence level showed that the parameters were significantly different in the two considered sets of samples, therefore indicating the presence of matrix effect due to the influence of FAMAE.

The presence of such matrix effect does not allow to use the same calibration curve to determine the analyte concentration in a gas oil with or without FAMAE. However, this is not a significant problem for casework. In fact, saponification number is not considered for classification purposes when FAMAE are present in fuel oils. In other words, the presence or absence of triacetin in FAMAE-containing heavy oil does not change the classification and taxation status of the product.

To verify the robustness of the method, measurements were repeated with a different number of scans and the calibration curve was constructed with the same standard with a different instrument. 8, 16, 32, 64 and 128 scans were used for the first set of experiments. Other instrumental parameters, such as resolution, apodization function, phase correction methods, etc. could in principle be changed through the software, but these are rarely if never modified in routine laboratory practice. On the other hand, it is common to act on the number of scans in order to improve spectral quality. Three replicates of the same standard containing 0.3% triacetin were acquired and the peak heights at 1218 cm^{-1} were measured and averaged. The relative uncertainty, assessed on the basis of the maximum semidispersion of the results was ⟨0.5% for 16 or more scans, whereas it increased to ⟩ 3% for 8 scans.

The calibration curves obtained with an instrument different from that originally used had different parameters than those reported in Table 1, albeit linearity was fully retained. This fact allows to say that the method is robust for a certain spectrophotometer, but not for different instruments: to perform a reliable analysis it is necessary to build a specific calibration curve for the instrument in use and only then
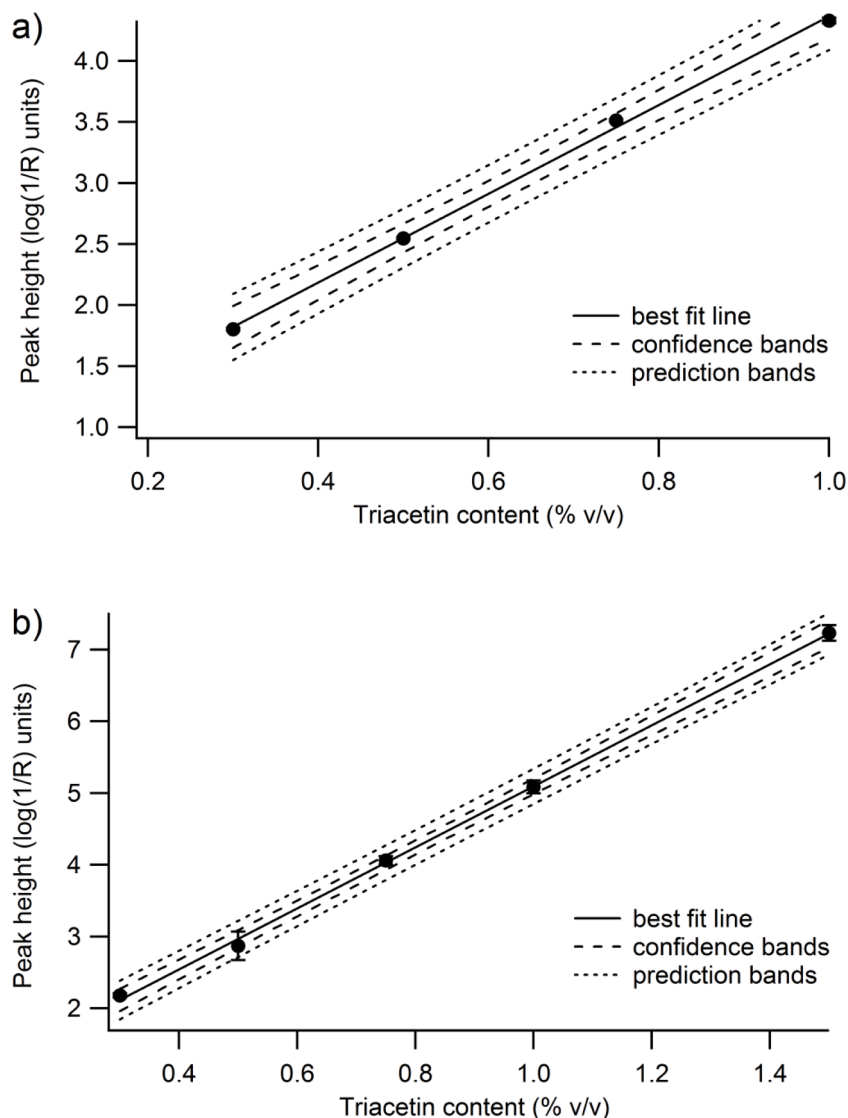
Fig. 4. Peak height of the 1218 cm^{-1} peak vs. triacetin concentration in (a) diesel non containing FAMAE and (b) diesel containing FAMAE. Error bars are shown for the peak heights in both cases. The very low relative error, of the order of magnitude of 0.5%, makes most of the error bars indistinguishable. Confidence and prediction bands are calculated at 95% confidence level.

Table 1
Calibration curve parameters for the lines shown in Fig. 4.

Matrix	Slope	Intercept	R^2
Diesel without FAMAE	3.74 ± 0.08	0.68 ± 0.05	0.999
Diesel with FAMAE	4.19 ± 0.04	0.92 ± 0.02	0.999

proceed with the reading of the sample.

The effect of temperature was not taken into account in studying robustness because this work was devised and carried out in a laboratory with rather constant ambient conditions and on a bench-top instrument. However, the effect of temperature and humidity may be significant in the case of field analyses and should be assessed when applying this method with portable instruments.

Another aspect which would deserve further studies is the effect on the quali- and quantitative analysis of triacetin of other contaminants, such as water, alcohols, aldehydes or other oxygenated organic compounds. Water was not considered in this work because its IR signals did not overlap with those of triacetin and because water content is normally very low (< 200 ppm, i.e. compliant with the limit set in the ISO

590 standard [1]) in casework samples. In fact, the presence of larger quantities of water would jeopardise the suitability of the designer fuel for use in diesel engines. On the other hand, studies are underway by some of the authors on complex designer fuels containing oxo oil derivatives.

Limit of detection (LOD) and limit of quantification (LOQ) were computed according the Eurachem guidelines. LOD was calculated as

$$LOD = 3 * s_0' = \frac{3 * s_0}{\sqrt{n}}$$

Where s_0 is the standard deviation of the blank at the frequency of interest and n is the number of measurements.

LOQ was obtained multiplying the standard deviation $s_{0'}$ by a factor of 8. The factor of 8 was chosen because at the chosen LOQ concentration, the standard deviation of 10 measurements of peak heights at 1218 cm^{-1} is low enough to consider the experimental data sufficiently accurate Table 2. shows the LOD and LOQ values for the two considered matrices.

In the region between LOD and LOQ it is only possible to state the presence of analyte, but quantification is impossible.

To verify the reliability of the quantification allowed by the proposed

Table 2
Limit of detection and limit of quantification of triacetin in the two considered matrices.

Matrix	LOD (% v/v)	LOQ (% v/v)
Diesel without FAMAE	0.1	0.3
Diesel with FAMAE	0.1	0.3

method, the concentration of 7 samples whose triacetin content was known were compared. Such samples did not contain FAMAE, so the calibration line for FAMAE-free matrices was used. As described in the experimental section, in the samples used for such comparisons the concentration of triacetin was determined gravimetrically on the polar extract. Albeit not extremely selective, such gravimetric method yields results significative enough to allow a correct classification of the product. The results are reported in Fig. 5.

As may be seen, the results are very well correlated. Gravimetry on the polar extract yields higher concentration values than IR spectroscopy. This was expected, because the polar extract on silica SPE cannot be entirely ascribed to triacetin. There can be in fact other components which may contribute to the polar fraction determined by the in house method described.

A further step for aiding a straightforward application of the results obtained in this work was taken correlating triacetin content with saponification number.

Fig. 6 shows that a correlation indeed exists. Part b) of Fig. 6 shows that extrapolating the data towards low concentrations, considering the confidence and prediction bands, 0.4% of triacetin is sufficient to increase saponification number of the fuel to a value > 4 mg KOH/g, misclassifying it as lubricant oil. A word of caution is necessary about a too extensive application of these data. Fig. 6 allows to estimate the saponification number of a diesel sample, but the width of confidence and prediction bands indicate that a significant uncertainty affects such estimate. Borderline estimates should always be verified by a direct determination of saponification number. The correlation in Fig. 6 must be refined with a larger number of samples, on a wider concentration and saponification number ranges. The use of casework samples will be required for such aim, in order to factor in the effects on saponification number of the multiple components contained in commercial diesels.

Finally, derived cetane number was measured for three samples,

containing 0.60, 0.61 and 0.8% v/v triacetin. Their cetane numbers were all in the interval 60–62, therefore confirming the suitability of triacetin-containing gas oil for use in diesel engines.

6. Conclusions

The aim of this work was to present a tool for fighting the growing problem of attempted tax fraud in petroleum products, in particular in diesel, and to provide a rapid and reliable method for the qualitative and semi-quantitative determination of glyceryl triacetate, i.e. triacetin, in this matrix. The results obtained show that indeed IR spectroscopy is a suitable technique for such objective. The speed and reliability of IR analysis will allow, for instance, an initial reading of samples directly in the field and an initial selection of "suspect" gas oils that will then require further analyses in the laboratory.

Designer fuels are a significant problem, and triacetin is not the only additive used for this kind of fraud. Further work is therefore needed to elucidate other strategies undertaken by criminal groups to make profit from misclassified petrochemicals.

CRediT authorship contribution statement

Rita Pastres: Data curation, Investigation, Writing – original draft. **Anna Luisa Panzeri:** Conceptualization, Writing – review & editing, Supervision, Project administration. **Doriana Visentin:** Conceptualization, Data curation, Investigation, Supervision, Validation, Methodology, Writing – review & editing. **Valerio Causin:** Supervision, Data curation, Methodology, Validation, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.
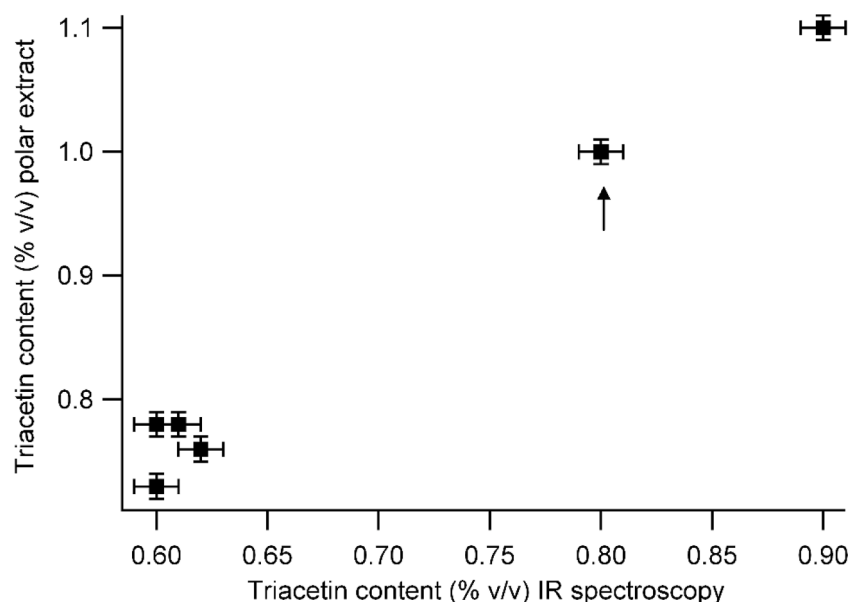

Fig. 5. Correlation between the concentration of triacetin in a set of seven samples, determined by two analytical methods. On the y axis the results of the determination of triacetin by gravimetry on the polar extract are reported. On the x axis, the concentration of triacetin by IR spectroscopy is reported. The arrow indicates two samples which yield superimposable data points.
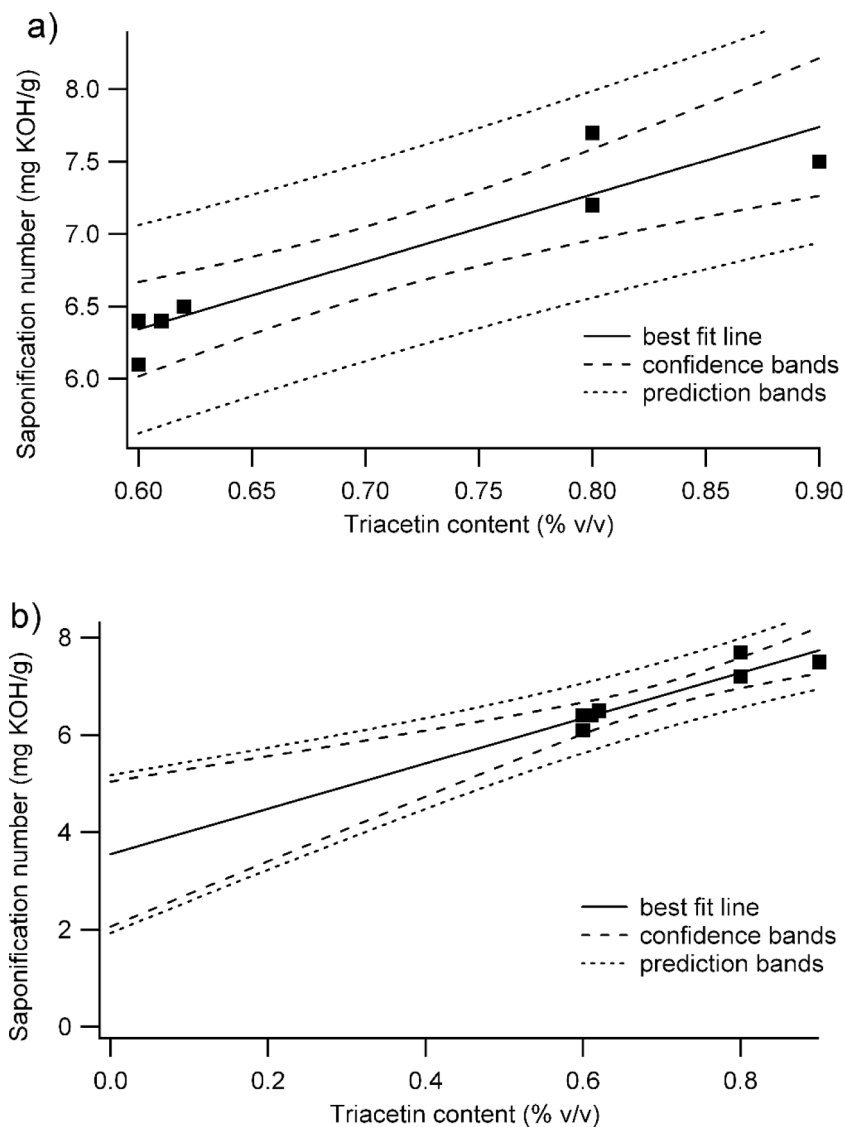
Fig. 6. (a) Saponification number vs. triacetin content determined by IR spectroscopy in a set of samples. Part b shows an extension of the best fitting line to low concentration values.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.talo.2022.100109.

References

[1] BS EN 590:2013+A1:2017 automotive fuels. Diesel. Requirements and test methods May 2017.

[2] Directive 2009/30/EC of the European Parliament and of the Council of 23 April 2009 amending directive 98/70/EC as regards the specification of petrol, diesel and gas-oil and introducing a mechanism to monitor and reduce greenhouse gas emissions and amending Council Directive 1999/32/EC as regards the specification of fuel used by inland waterway vessels and repealing directive 93/12/EEC, Off. J. Eur. Union L 140 (2009). June 5.

[3] Council regulation (EEC) No 2658/87 of 23 July 1987 on the tariff and statistical nomenclature and on the common customs tariff, Off. J. Eur. Union L 256 (1987). September 7 with subsequent integrations and amendments.

[4] ASTM D94-07 (2017). Standard test methods for saponification number of petroleum products. July 10, 2017.

[5] Commission implementing regulation (EU) 2018/125 of 24 January 2018 amending annex I to council regulation (EEC) No 2658/87 on the tariff and statistical nomenclature and on the common customs tariff, Off. J. Eur. Union L 22/10 (2018). January 26.

[6] A. Casas, J.R. Ruiz, M.J. Ramos, A. Perez, Effects of triacetin on FAMAE quality, Energy Fuels 24 (2010) 4481–4489.

[7] S.M. Dhawan, S.C. Barton, D.G. Yadav, Interesterification of triglycerides with methyl acetate for the co-production FAMAE and triacetin using hydrotalcite as a heterogenous base catalyst, Catal. Today 375 (2021) 101–111.

[8] R. Jatoth, S.K. Gugulothu, G.R.K. Sastry, Experimental study of using FAMAE and low cetane alcohol as the pilot fuel on the performance and emission trade-off study in the diesel/compressed natural gas dual fuel combustion mode, Energy 225 (2021) art. no. 1201218.

[9] D. Spataru, A.P.S. Dias, L.F.V. Ferreira, Acetylation of FAMAE glycerin using glycerin and glucose derived catalysts, J. Clean. Prod. 297 (2021) art. no. 126686.

[10] P. Mukhopadhyay, R. Chakraborty, LCA of sustainable FAMAE production from fried Borassus flabellifer oil in energy-proficient reactors: impact assessment of multi fuel-additives on pour point, NOx and engine performance, Sustain. Energy Technol. Assess. 44 (2021) art. no. 100994.

[11] D. Unlu, N.D. Hilmioglu, Production of fuel bioadditive "triacetin" using a phosphomolybdic-acid-loaded PVA membrane in a pervaporation catalytic membrane reactor, Energy Fuels 33 (2019) 2208–2218.

[12] A. Zare, T.A. Bodisco, M.N. Nabi, F.M. Hossain, Z.D. Ristovski, R.J. Brown, A comparative investigation into cold-start and hot-start operation of diesel engine performance with oxygenated fuels during transient and steady-state operation, Fuel 228 (2018) 390–404.

[13] A. Zare, M.N. Nabi, T.A. Bodisco, F.M. Hossain, M.M. Rahman, T.C. Van, Z.D. Ristovski, R.J. Brown, Diesel engine emissions with oxygenated fuels: a comparative study into cold-start and hot-start operation, J. Clean. Prod. 162 (2017) 997–1008.

[14] A. Zare, T.A. Bodisco, M.N. Nab, F.M. Hossain, Z.D. Ristovski, R.J. Brown, Engine performance during transient and steady-state operation with oxygenated fuels, Energy Fuels 31 (2017) 7510–7522.

[15] A. Zare, M.N. Nabi, T.A. Bodisco, F.M. Hossain, M.M. Rahman, Z.D. Ristovski, R. J. Brown, The effect of triacetin as a fuel additive to waste cooking FAMAE on engine performance and exhaust emissions, Fuel 182 (2016) 640–649.

[16] C.V. Lacerda, M.J.S. Carvalho, A.R. Ratton, I.P. Soares, L.E.P. Borges, Synthesis of triacetin and evaluation on motor, J. Braz. Chem. Soc. 26 (2015) 1625–1631.

[17] S. Amma, J. Luo, S.H.Kim C.G.Pantano, Specular reflectance (SR) and attenuated total reflectance (ATR) infrared (IR) spectroscopy of transparent flat glass surfaces: a case study for soda lime float glass, J. Non Cryst. Solids 428 (2015) 189–196.

[18] V. Causin, C. Marega, G. Guzzini, A. Marigo, Forensic analysis of poly(ethylene terephthalate) fibers by infrared spectroscopy, Appl. Spectrosc. 58 (2004) 1272–1276.

[19] ASTM D7170-16 standard test method for determination of derived Cetane number (DCN) of diesel fuel oils—fixed range injection period, constant volume combustion chamber method. October 1, 2016.

[20] F. Chinnici, E. Durán Guerrero, F. Sonni, N. Natali, R. Natera Marín, C. Riponi, Gas chromatography−mass spectrometry (GC−MS) characterization of volatile compounds in quality vinegars with protected european geographical indication, J. Agric. Food Chem. 57 (2009) 4784–4792.